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Cryomilled aluminum alloys are being developed for aerospace applications. These high-strength aluminum alloys are currently targeted for low-temperature rocket applications. This research was focused on the fundamental strengthening mechanisms in these alloys. The temperature dependence of strength and ductility was investigated. The theoretical models predict strengthening at low temperatures and decrease of flow stress with decreasing grain size because of diffusional flow and grain boundary sliding at elevated temperatures. Results from this study show discrepancies with theoretical models. Significant work hardening was observed at room temperature in both Al-Ti-Cu and Al-Mg alloys. The strain hardening exponents suggest contributions from both direct and indirect work hardening mechanisms. At elevated temperatures, the stress-strain rate data for Al-Mg alloys can be best described by a power law constitutive relationship with a stress exponent of 5 and threshold stress. However, the activation energy is very high even after compensating for threshold approach. This indicates that dissolution of Al_3Mg_2 precipitates influence the deformation kinetics.

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FUNDAMENTAL INVESTIGATIONS OF PLASTICITY IN HIGH STRENGTH NANOSTRUCTURED ALUMINUM ALLOYS
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Final Report

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Abstract

Cryomilled aluminum alloys are being developed for aerospace applications. These high-strength aluminum alloys are currently targeted for low-temperature rocket applications. This research was focused on the fundamental strengthening mechanisms in these alloys. The primary sources of strengthening in cryomilled aluminum alloys are dispersion of nanocrystalline particles and ultrafine grain size that form during the cryomilling stage. Optimized thermo-mechanical processing of cryomilled powder results in an ultrafine grain size material with nanocrystalline particles. We investigated the temperature dependence of strength and ductility. The theoretical models predict strengthening at low temperatures and decrease of flow stress with decreasing grain size because of diffusional flow and grain boundary sliding at elevated temperatures [1]. Our results show discrepancies with theoretical models. Significant work hardening was observed at room temperature in both Al-Ti-Cu and Al-Mg alloys. The strain hardening exponents suggest contributions from both direct and indirect work hardening mechanisms. At elevated temperatures, the stress-strain rate data for Al-Mg alloys can be best described by a power law constitutive relationship with a stress exponent of 5 and threshold stress. However, the activation energy is very high even after compensating for threshold approach. This indicates that dissolution of Al_3Mg_2 precipitates influence the deformation kinetics.

Research Objectives

High-strength cryomilled aluminum alloys are being developed for aerospace applications. The temperature dependence of the flow stress, ductility, and creep behavior is considered very important for the identification of application range of these alloys. This study was focused on the fundamental strengthening mechanisms in high-strength nanostructured aluminum alloys. The important issues are: (a) influence of thermo-mechanical processing route on microstructure, (b) influence of microstructure on intermediate temperature ductility and deformation mechanisms, (c) kinetics of dislocation movement and work hardening in ultrafine grained materials, and (d) nature of dislocation-particle interaction at various temperatures and related change in strength.

Approach

To investigate these issues, we adopted a combination of transmission electron microscopy (TEM), thermal treatments, and mechanical testing methods. Two different alloy system, Al-Mg and Al-Ti-Cu types, and four different thermomechanical processing routes, multiaxial forging, rod extrusion, tube extrusion and friction stir processing (FSP), were chosen in consultation with Boeing, Rockwell Scientific and AFRL (Table I). We conducted tensile tests, on materials supplied by Boeing and Rockwell Scientific with different thermomechanical processing, in the temperature range of -252.7-400 °C in the strain rate range of 10^{-4}-10^{-1} s^{-1}. To obtain direct information on dislocation generation, movement, and annihilation, we tried a few in-situ TEM straining experiments, which were not successful. The fundamental understanding of thermal stability of microstructure and deformation processes at various temperatures will help in the optimization of microstructure and processing conditions. The processing optimization and the choice of alloy systems was done by the collaborators; the Boeing Company (C. C. Bampton and D. Matejczyk), the Rockwell Scientific Company (P. B. Berbon), and the Air Force Research Laboratory (S. L. Semiatin), where significant effort is currently underway to develop 'second-generation' nanophase-Al alloys. This research directly complemented this broad effort and was focused on the fundamental aspects of deformation. This was an ideal University-AFRL-Industry collaboration in the development of high-strength aluminum alloy for space and aircraft applications.

Table I. A summary of microstructural features of nanophase aluminum alloys.

Alloy	Processing Route	Average grain size, nm	Average particle size, nm	Particle volume fraction, %
Al-7.5 Mg	Forged block	774	Al_3Mg_2-206; fine particles- 32	-**
Al-8.4 Mg	Extruded tube	685	Al_3Mg_2-168; fine particles- 12	-
Al-6.7 Mg	Extruded bar	782	Al_3Mg_2-140; fine particles- 15	-
Al-10 Ti-2 Cu	Extruded rod	349	116	21.7
Al-10 Ti-2 Cu	FSP plate	516	122	21.7

** Quantification will be done after identification of various types of precipitates.

Research Accomplishments

Only the Key Accomplishments are highlighted in this final report. Further details were reported in previous annual reports[2,3] and can be provided by the PI.

1. Identification of N Bearing Particles

When this project started in 1999, a major question was identification of N bearing particles in materials processed at Boeing and Rockwell Science Center.. Figure 1 shows N containing particle with EELS spectra. In addition, we have observed 15-20 nm disc shaped particles in some grains. Although these particles have not yet been identified in this system, Susegg et al. [4] identified these particles as AlN in cryomilled Al alloy.

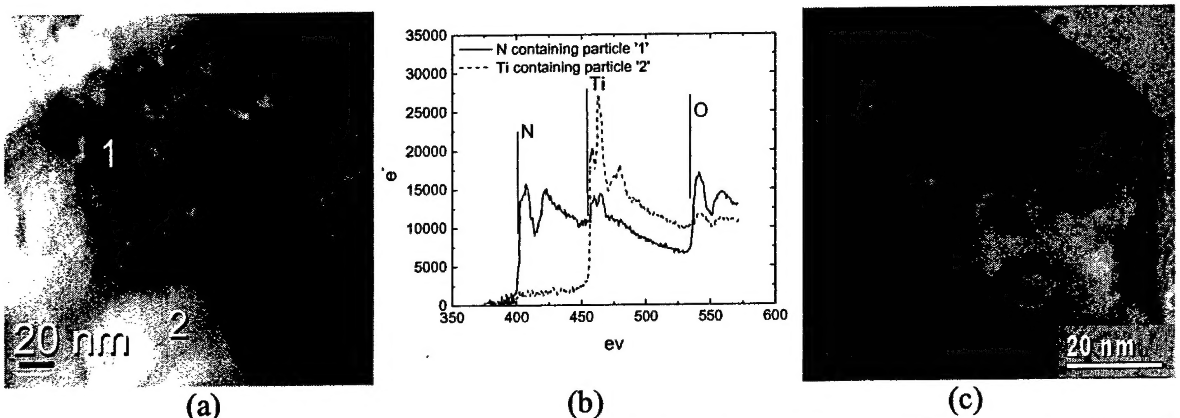

Figure 1. (a) A STEM micrograph of FSP Al-Ti-Cu alloy, (b) two EELS spectra from N containing particle marked '1' and Ti containing particle marked '2', and (c) a bright field TEM micrograph showing disc shape particles in edge-on condition.

2. Temperature Dependence of Deformation Mechanism

For ultrafine grained materials, the deformation mechanism is likely to change. An important issue is the relative contribution of dislocation and diffusional deformation mechanisms. Some specific issues that we focused on were, (a) extent of work hardening and its temperature dependence, and (b) change in deformation mechanism with temperature. Figure 2 shows flow curves for current nanophase aluminum alloys at room temperature and a few nanostructured metals from literature [5-7]. It can be noted that a general trend does not exist in ultrafine grained and nanocrystalline materials. The nanophase aluminum alloys exhibit significant work hardening in certain processed conditions. Work hardening characteristic is considered very important for engineering applications to avoid catastrophic failures. Hazzledine and Louat [8] have discussed the relative contributions of direct (particle) and indirect (grain boundary) work hardening in dispersion strengthened materials. According to their model, the normal flow stress is given by

$$\sigma = MG\left[\left(\frac{6fb}{\pi^2 r}\right)^{1/2} + \left(\frac{\alpha^2\varepsilon^2}{C^2} + \frac{6\alpha^2 f\varepsilon b}{r}\right)^{1/2}\right] \tag{1}$$

where M is the Taylor factor of about 3, G is the shear modulus, b is the Burgers vector, α is a constant equal to ¼, ε is the strain and C is a constant of about 70. The second and third terms on the right hand side in equation (1) represent hardening from forest dislocation intersections and grain boundary intersections. Hardening increment due to dislocation accumulation at particles contributes equally to the third term. The change in work hardening exponent with temperature is shown in Figure 2b for Al-Ti-Cu and Al-Mg alloys. It is interesting to note that Al-Mg alloy shows work hardening exponents of >0.5 at lower temperatures. The implication is that the forest dislocation interactions contribute significantly in Al-Mg alloys, but not in Al-Ti-Cu alloys. In high Mg containing alloys, where the Mg is in solid solution, the plastic deformation behavior can be affected by dynamic strain aging and we have observed serrated flow in the beginning of flow curve. Another important observation from the present work is that processing influences the work hardening characteristics significantly and in turn the overall ductility of the material. Extruded Al-Mg tubes and FSP Al-Ti-Cu alloys show the best characteristics.

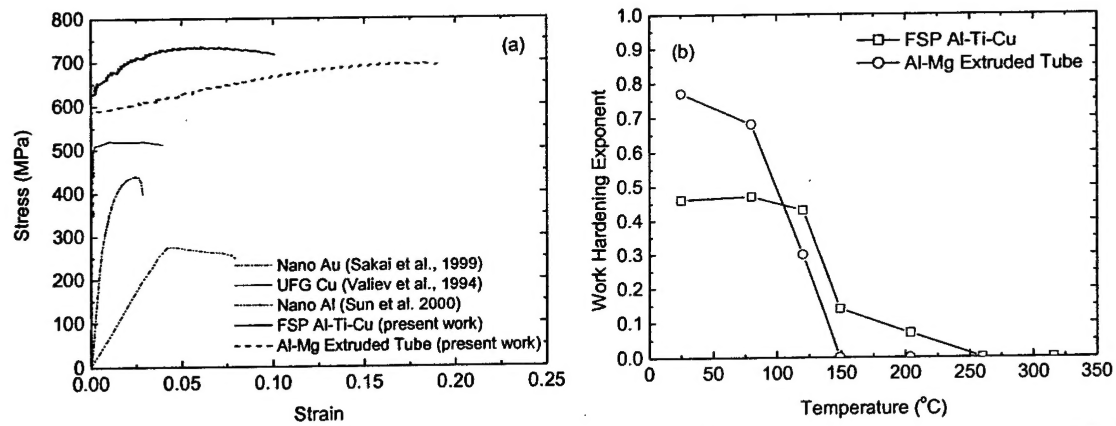

Figure 2. (a) Flow curves for nanophase aluminum alloys at room temperature. (b) The variation of work hardening exponent with temperature for Al-Ti-Cu and Al-Mg.

To establish the strain rate dependence of flow behavior, we conducted tests at several temperatures for both Al-Mg and Al-Ti-Cu alloys. The results of Al-Mg extruded tube material is shown in Figure 3(a). At room temperature, the flow stress is independent of strain rate. At elevated temperature the apparent stress exponent varies from 7 to 10. It is indicative of dislocation mechanism. We have analyzed the data using the dislocation climb mechanism with a threshold stress. The normalized strain rate is plotted against normalized stress in Figure 3(b). The correlation in Figure 3(b) gives a constitutive relationship for deformation of Al-Mg alloy as,

$$\dot{\varepsilon} = 7.58\times10^{26}\,\frac{D_o G b}{kT}\exp\left(-\frac{252{,}000}{RT}\right)\left(\frac{\sigma-\sigma_o}{E}\right)^5 . \tag{2}$$

The activation energy for Al-Mg alloy is significantly higher than the activation energy of 142 kJ/mol for Al self diffusion or for lattice diffusion of Mg in Al. The present alloy contains 8.4 Mg. Mg is present in both solid solution and Al_3Mg_2 precipitates. In the temperature range of 205-260 °C, the solubility of Mg changes from 3.5 to 5.2 wt.%. The decrease in the volume fraction of precipitates will lead to lower strength and therefore the apparent activation energy will be higher.

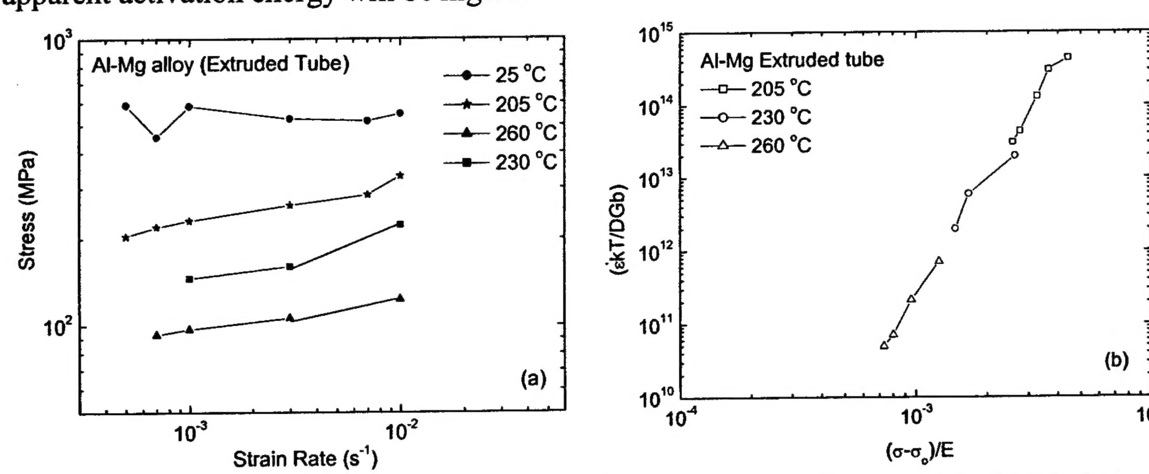

Figure 3. (a) Stress-strain rate behavior of Al-Mg extruded tube material. (b) Variation of normalized strain rate with normalized stress.

The constitutive relationship for Al-Ti-Cu alloy was,

$$\dot{\varepsilon} = 4\times10^{16}\,\frac{D_oGb}{kT}\,exp\left(-\frac{145{,}000}{RT}\right)\left(\frac{\sigma-\sigma_o}{E}\right)^5 \qquad (3)$$

Note the difference in the kinetics constant and the activation energy. The microstructure of this alloy does not change with temperature within the investigated range. We believe that this thermal stability leads to the activation energy that is close to the lattice self-diffusion of aluminum. This indicates that dislocation-particle interaction within the grain is the rate controlling step. Also, the kinetics is different in Al-Mg and Al-Ti-Cu alloy. Figure 4 shows the threshold stress variation with temperature. The theoretical prediction based on the positive climb concept is shown in Figure 4(b).

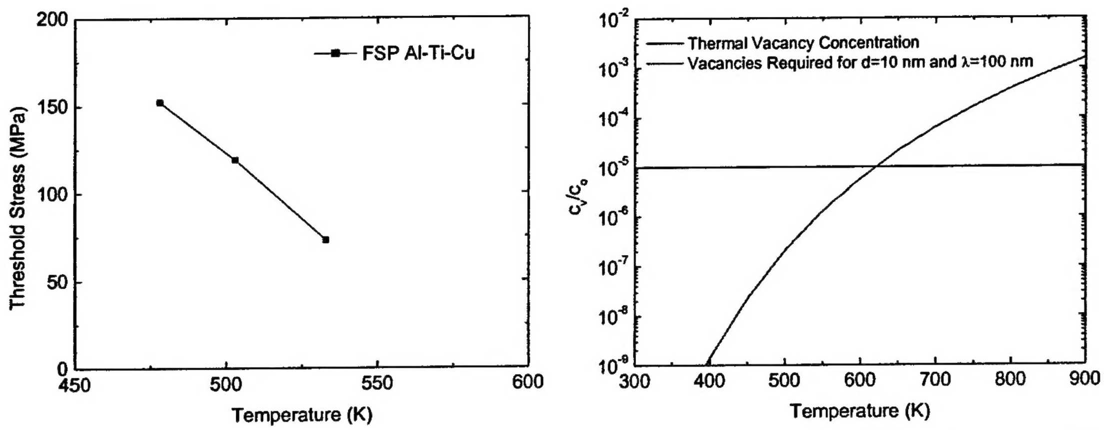

Figure 4. (a) The variation of threshold stress with temperature for Al-Ti-Cu alloy. (b) The theoretical prediction of positive climb concept can qualitatively account for this variation. The model needs to be developed further for quantitative explanation.

3. Intrinsic vs Flaw-dependent Deformation Behavior

A major contribution of this study was the contribution to highlight intrinsic vs flaw dependent behavior using friction stir processing. Friction stir processing was used for the first time to homogenize the microstructure. This resulted in intrinsic behavior of material, i.e. flaw dependent material deformation did not mask the actual behavior of material. Figure 5 highlights this by comparing the tensile and compressive behavior. It can be noted that for the FSP material, tensile and compressive UTS values are similar, whereas the values for extruded material are different. In the extruded material, the tensile test data reflect the flaw-dependent behavior. Such results should not be used for fundamental understanding of deformation mechanisms.

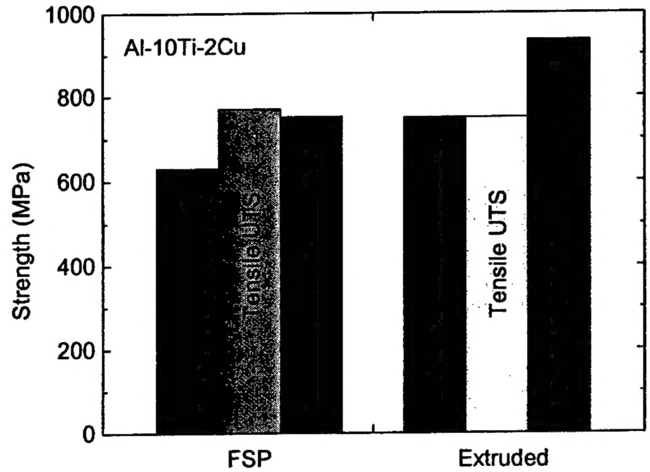

Figure 5. A comparison of tensile and compressive deformation results for Al-Ti-Cu alloy.

4. Intermediate Temperature Ductility

When we started this project, a key issue for powder metallurgy processed aluminum alloy was the intermediate temperature ductility. Our results (Figure 6) proved that the intermediate temperature ductility depends on processing and not on chemistry of the alloy.

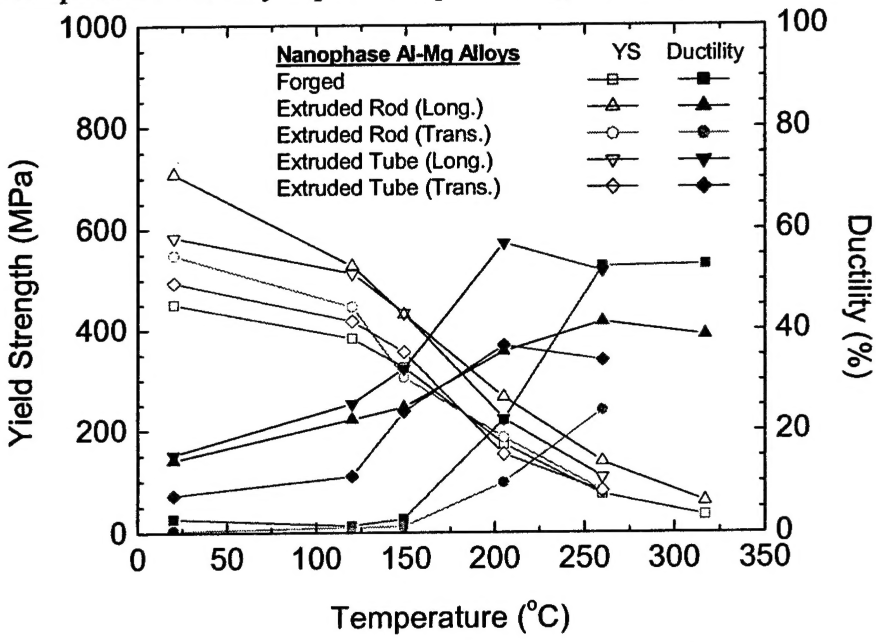

Figure 6. Variation of strength and ductility with temperature. Note that the intermediate temperature ductility depends on the processing route.

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the U.S. Government. This work was carried out in collaboration with C. C. Bampton and D. Matejczyk of the Boeing Company, P. B. Berbon of the Rockwell Scientific and S. L. Semiatin of the Air Force Research Laboratory. The supply of nanophase aluminum alloys and very useful discussions with the collaborators are gratefully acknowledged. The efforts of Jing Zheng, graduate student, are also acknowledged. The analytical TEM work was carried out with the help of Dr. Ray Twetsen in the Center for Microanalysis of Materials, University of Illinois, which is partially supported by the U.S. Department of Energy.

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Personnel Supported

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